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Pressure Effects in Environmental Studies

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Recent advances in MEMS based instrumentation for transmission electron microscopy has paved the way for *in situ* investigation of materials with unprecedented spatial resolution under high pressures and elevated temperatures. Combined with traditional differentially pumped environmental TEMs, researchers can investigate phenomena such as the melting of nanoparticles, growth of individual graphene layers [1], nanowire [2] and nanotube growth and surface structure of catalytic materials exposed to pressures in the range spanning from 10^{-8} Pa to atmospheric pressure all at a resolution in the Ångström regime. In addition, newly developed image acquisition equipment, CMOS based and direct electron detection, provides the ideal platform for analyzing the dynamics of nanostructured materials [3].

Iron oxides play a significant role Fischer-Tropsch synthesis. A specific topic of debate has been the active phase and how to achieve it. Typically, these catalysts start out as iron oxides (Fe_2O_3). These oxides are then reduced to Fe_3O_4 and in some cases all the way to metallic iron. After reduction, the sample is carburized to the active iron carbide state. The ideal conditions for these stages is not fully understood and is still a subject of investigation.

Here we present an initial investigation of the reduction stage of iron oxide, Fig. 1, using pressures ranging from what can be realized in an ETEM (~ 1 -5 mbar) using a DENSsolutions Wildfire heating holder and up to atmospheric pressure using the DENSsolutions Climate holder. Even at high pressures of H_2 , the resolution is maintained in the climate holder compared to the lower pressures of the ETEM and the phase of the sample can be determined.

Here we will show the results of these initial investigations along with some initial findings on and prospects of working with high-pressure cells in the TEM and analysis of dynamic data.

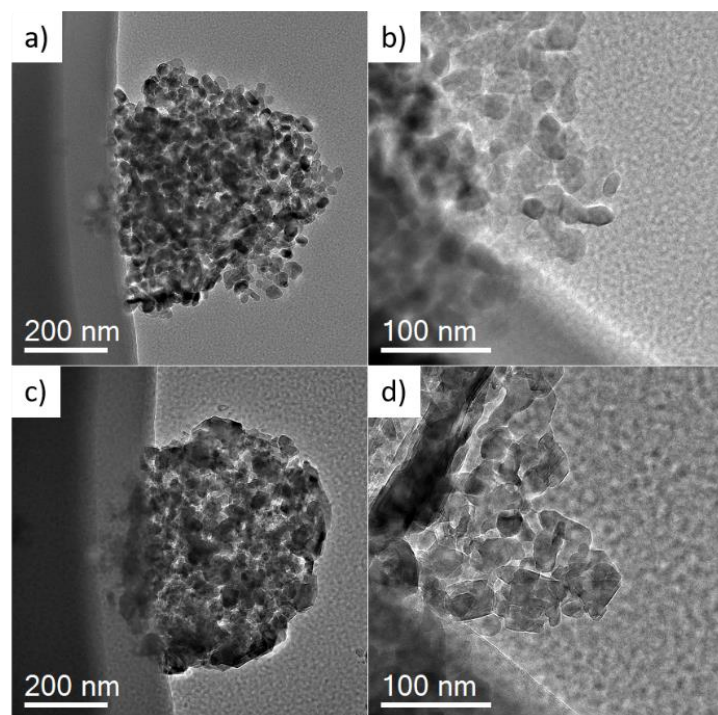


Figure 1: Fe_2O_3 imaged in vacuum at 150°C (a and b) and after 2 hours at 700°C in 825 mbar H_2

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